



82966LMB
Customer No. 01333

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Christine J. Landry-Coltrain, et al

SMALL POROUS POLYESTER
PARTICLES FOR INKJET USE

Serial No. 10/028,130

Filed 20 December 2001

Commissioner for Patents
P.O. Box 1450
Alexandria, VA. 22313-1450

Group Art Unit: 1774

Examiner: Pamela R. Schwartz

I hereby certify that this correspondence is being deposited today with the United States Postal Service as first class mail in an envelope addressed to Commissioner For Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

Christine Tolhurst

Christine Tolhurst

November 23, 2004

Date

SECOND DECLARATION UNDER RULE 132

1. I, Christine J. Landry-Coltrain, state that I am a resident of Fairport, N.Y., in the county of Monroe and am a citizen of the United States. I obtained a Bachelor of Science degree in McGill University from Montreal, Canada in 1980. I also have a Ph.D. degree from the University of Wisconsin in Madison Wisconsin in 1985, with a focus on polymer science. I have been an employee of Eastman Kodak Company (hereinafter referred to as Kodak) since 1985. I have been assigned to work in research and development in areas relating to polymer science, such as polymer blends and composites, and media development, such as inkjet and thermal media, and studies relating to the physical properties of polymers.
2. I am one of the co-inventors of U.S. Serial No. US 10/028,130.
3. I have read the Office Action issued on July 29, 2004 and I am familiar with the references cited therein.

4. The following particles evidences the functionality of the present invention utilizing different materials contained in the category of polyesters that is now claimed.
5. Two bead preparations were made: PE-12: Small beads of invention and PE-11: Large beads not of invention
6. Preparation of Large beads (PE-11):

An organic phase consisting of 90.00 g Fineclad® 385 (a nonionic, oil-soluble, unsaturated aliphatic polyester resin obtained from Reichhold Co.), 7.2 ml hexadecane, 1.80 g AIBN, 45.0 g divinylbenzene (mixture of *m* and *p* isomers, 80% with remainder being ethylstyrene) and 45.0 g chloromethylstyrene (mixture of *m* and *p* isomers) dissolved in 60180.0 g toluene was prepared. An aqueous phase consisting of 14.4 g dodecanethiol-endcapped acrylamide oligomer (prepared exactly as described in U.S. Patent No. 6,127,453, col. 9, lines 40-55) dissolved in 1080.0 g deionized water was combined with the organic phase in a 2L beaker and the mixture was homogenized for 10 minutes using a Silverson L4R mixer at the highest speed setting. The resulting dispersion was transferred to a 3-neck round bottom flask outfitted with a condenser, nitrogen inlet, and mechanical stirrer, bubble degassed with nitrogen for 10 minutes, and was heated for 16 hours at 70°C in a constant temperature bath under a positive pressure of nitrogen. 26.3 g N,N-dimethylethanolamine was added and the dispersion was allowed to stir at 80°C for 24 hours. The resulting dispersion of quaternized particles was subjected to rotary evaporation until the collected condensate came over as one phase. The dispersion was filtered through four ply cheesecloth and was purified further by diafiltration with 8 volumes of water through a 100K cutoff membrane using a Millipore Amicon® ultrafiltration apparatus and concentrated to 21.3% solids. The mean particle size of the washed dispersion was determined to be 3.26 µm (CV = 40.04%, Proportion = 100 %) using a Horiba LA-920® particle size analyzer.

7. Small beads (PE-12)

The same aqueous and organic phases described for PE-11 were prepared and combined. The combined phases were emulsified by a two-step process consisting homogenization for 5 minutes using a Silverson L4R mixer followed by passage twice through a microfluidizer. The emulsified reaction mixture was transferred to a 3-neck round bottom flask outfitted with a condenser, nitrogen inlet, and mechanical stirrer, bubble degassed with nitrogen for 10 minutes, and was heated for 16 hours at 70°C in a constant temperature bath under a positive pressure of nitrogen. 26.3 g dimethylethanolamine was added and the reaction was stirred overnight at 70°C. The resulting dispersion of quaternized particles was subjected to rotary evaporation until the collected condensate came over as one phase. The dispersion was filtered through four ply cheesecloth and was purified further by dialysis for 16 hours using 12-14K cutoff dialysis tubing. The resulting dispersion was freeze-dried to obtain 142.5 g of a fine white powder which was easily redispersible in water. The particle size was found to be 0.371 μM (CV = 29.11%, Proportion = 100 %) using a Horiba LA-920® particle size analyzer.

8. Preparation of Element using P-12 small invention beads of Fineclad polyester

A coating composition was prepared from 10.2 wt. % of porous particles PE-12, 1.8 wt. % poly(vinyl alcohol), PVA, (Gohsenol® GH-17 from Nippon Gohsei Co.), and 88 wt. % water. [The relative proportions of porous polyester particle to PVA are therefore 85/15 by weight]. The solution was coated onto a base support comprised of a polyethylene resin coated photographic paper stock, which had been previously subjected to corona discharge treatment, using a calibrated coating knife, and dried to remove substantially all solvent components to form the ink receiving layer. The thickness of the dry ink receiving layer was measured to be about $25 \pm 2 \mu\text{m}$.

9. Preparation of Control Element using P-11 large beads of Fineclad polyester
A coating composition was prepared from 71.76 wt. % of the PE-11 aqueous dispersion, 2.7 wt. % poly(vinyl alcohol), PVA, (Gohsenol® GH-17 from Nippon Gohsei Co.), and 25.53 wt. % water. [The relative proportions of porous polyester particle to PVA are therefore 85/15 by weight]. The solution was coated onto a base support comprised of a polyethylene resin coated photographic paper stock, which had been previously subjected to corona discharge treatment, using a calibrated coating knife, and dried to remove substantially all solvent components to form the ink receiving layer. The thickness of the dry ink receiving layer was measured to be about $38 \pm 2 \mu\text{m}$.
10. Gloss Assessment of Coated Elements

| Element | Cyan D-max | 60° gloss | Mean diameter (micron) | Proportion (%) |
|-----------------|------------|-----------|---------------------------|----------------|
| P-12 | 1.9 | 47 | 0.37 | 100 |
| Control P-11 | 1.4 | 2.4 | 3.3 | 100 |

The above results show that high surface gloss for the element containing porous polyester particles can only be achieved with the incorporation of porous polyester particles that have a mean diameter of less than 0.5 micrometers, compared to the control elements having only porous polyester particles that have a mean diameter of greater than 0.5 micrometers.

11. I further declare that all statements made herein of my own knowledge are true and that the statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent resulting therefrom.

Date: Nov-19-2004

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Christine Tolhurst

Christine Tolhurst

November 23, 2004


Date

SECOND DECLARATION UNDER RULE 132

1. I, Jeffrey W. Leon, state that I am a resident of Rochester, N.Y., in the county of Monroe and am a citizen of the United States. I obtained a Bachelor of Science degree in Chemistry from State University of New York (SUNY) in Albany, NY in 1989. I was an employee of Schenectady Chemicals in Schenectady, New York in 1989 as a resins chemist. I received a PhD in Chemistry from the University of Rochester, Rochester, NY in 1994, relating to photodegradation of polyester coatings. I did Postdoctoral work at Cornell University, in Ithaca, New York, from 1994-1996, relating to synthesis of dendrimeric polymers. I have been an employee of Eastman Kodak Company (hereinafter referred to as Kodak) since 1996. I have been assigned to work in research development relating to synthesis of latex polymer particles and beads, thermoreactive polymers, inkjet receiver media, polyester synthesis, and water-borne coatings.
2. I am one of the co-inventors of U.S. Serial No. US 10/028,130.
3. I have read the Office Action issued on November 19, 2003 and am familiar with the references cited therein.

4. For the method of preparation of the polyester-containing particles of this invention, different emulsification methods are used. It is the choice of emulsification method, which, of all of the reaction parameters, most strongly influences the resulting particle size. Hence different emulsification methods are commonly used to make particles which differ in particle size.
5. Higher shear methods, such as microfluidization, will typically give smaller particles than, for example, homogenization using a Silverson Mixer.
6. After emulsification, a dispersion results which consists of droplets of the ethylenically unsaturated monomer(s) (in this case, divinylbenzene), the polyester, and the porogenic solvent (in this case, toluene). As the crosslinking reaction is initiated, the droplet becomes a porous microsphere. The composition of the porous microsphere would not be expected to be different if it was formed from a smaller or larger droplet.
7. The microsphere should be the same regardless of the type of emulsification used except that the size will be different. Any scientist trained in the art would expect the composition and porous morphology of the particles to be the same regardless of the type of emulsification used. Only the particle size is affected.
8. To achieve the variety of particle sizes necessary to evaluate the present invention, different emulsification techniques were utilized.
9. I further declare that all statements made herein of my own knowledge are true and that the statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent resulting therefrom.

Date: 11/18/04


Jeffrey W. Leon